# PATENT SPECIFICATION

DRAWINGS ATTACHED

**876,070**

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## An improved countercurrent extraction apparatus.

COMPLETE SPECIFICATION

We, F. HOFFMAN-LA ROCHE & CO., AKTIENGESELLSCHAFT, a Swiss Company of 124-184 Grenzacherstrasse, Basle, Switzerland, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

This invention relates to an improved extraction apparatus intended for countercurrent extractions involving substantially immiscible liquids. More particularly, the invention relates to an improved extraction column for countercurrent solvent extraction (that is to say, for extraction processes involving the transfer of a solute or solutes from one or more solvents to another solvent or solvents which is or are substantially immiscible with the original solvent or solvents; also sometimes referred to as countercurrent liquid extraction) and fractional liquid extraction (that is to say, for extraction processes in which two mutually immiscible liquids are added to a solution and each preferentially dissolves a different component from that solution).

It is conventional practice to extract or separate a solute or solutes from a solution using two substantially mutually immiscible solvents flowing countercurrently in intimate contact with one another and the solution containing the solute or solutes in a tower or column. Purification is effected through the selective solubility of the solute or solutes in the solvents. Current work in the field stresses intensive agitation of the liquids in the column. This is usually accomplished by means of a plurality of rapidly rotating fins or paddles. Agitating devices with jet action have also been recommended in order to increase the subdivision of the liquids. Baffles are usually inserted in the column in order that the liquids follow a long and circuitous path. They also provide calming zones where the liquid droplets formed as the disperse phase in the agitation zones may coalesce between periods of agitation. Packing, such as wire mesh or steel wool, is also frequently utilized to promote coalescence of the droplets. These expedients increase the residence time of the liquids in the column and so limit the amount of liquid which can be passed through in a given time, thus reducing the column capacity.

It has now been found, according to the present invention, that efficient countercurrent solvent and fractional liquid extraction can be achieved using a column in which the agitation is effected by means of a plurality of open network reciprocating discs and which is not povided with baffles, packing or other impediment to the flow of liquids.

The extraction apparatus provided by the invention comprises a vertical elongated unbaffled column having inlets and outlets for introduction and removal of liquids, a vertical centrally located movable shaft adapted to reciprocate in a vertical direction, a plurality of horizontal open network agitating discs mounted on said shaft, each of said discs extending over substantially the entire interior cross-section of the column while leaving room for the passage of liquid between the periphery thereof and the interior wall of the column and containing large openings together comprising 50% to 65% of the cross-sectional area of the disc, and a reciprocating drive mechanism for said shaft.

In the apparatus provided by the invention, the agitating discs have large openings and there are no seals between the discs and column wall. Resistance to the flow of liquids is minimized, yet contrary to expectations effiecient mixing is still maintained. Substantially unimpeded flow through the column is thus ensured for the liquids and a high rate of passage is achieved. The apparatus provides continuous passage for

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the liquids. It is particularly adapted for viscous liquids. Moreover, the height of a theoretical stage is independent of the diameter of the column for any given extraction system and for a given spacing of the agitating discs.

According to the present invention, the vertical shaft adapted to reciprocate in a vertical direction is centrally located within the elongated column having no other impediment to the flow of liquids, such as baffles or packing. The shaft is mounted in suitable bearings at or near the top and bottom of the column so that movement of the shaft is restricted to a vertical direction. A reciprocating drive mechanism is externally connected to the shaft to provide the motion. The drive mechanism may, if desired, be equipped to vary the speed and amplitude of the oscillations.

Mounted at spaced intervals on the shaft along the longitudinal axis of the column and placed perpendicular to that axis are a plurality of open network agitating discs. These discs comprise thin plates in which large openings form the open network. The horizontal, open network discs extend substantially completely over the cross section of the column and generally conform in outline to the interior wall of the column. They are, however, slightly smaller in cross-sectional area than the column interior so that there is no contact or seal between the peripheral edges of the discs and the interior wall of the column and liquid may flow between the discs and the interior wall. The openings in the discs are large in size and make up 50% to 65% of their cross-sectional area. It is essential that the openings in the discs be relatively large rather than a larger number of small openings; that is to say, the number of openings should preferably be set at a minimum consistent with the requirements of disc strength. The large openings in the discs and the lack of contact between discs and column wall avoid the collection of liquid drops beneath the discs during agitation and thereby introduce little resistance to the flow of the liquids without sacrifice of efficiency. The reciprocating motion translated to the agitating discs through the shaft breaks up into drops the liquid flowing as the disperse phase in the column.

The agitating discs may be constructed of any structurally adequate material such as metal or plastic. According to a preferred embodiment, each agitating disc comprises a latticework of expanded metal as illustrated in Figure 2. Alternatively, a spoked wheel-like agitator as shown in Figure 3 may be used. The latter is particularly adapted to be stamped out of plastic. There is no packing or seal between the periphery of the agitating discs and the interior surface of the column. When metal discs are used in glass columns, however, it may be desirable to avoid accidental metal-to-glass contact resulting from any flexing of the shaft by providing a limited number of bearing surfaces or bumpers, consisting of plastic, for example, at spaced points on the peripheries of some of the discs. The spacing of the discs on the shaft may be varied according to the kind of extraction to be undertaken. In general, the discs may be spaced more closely in those parts of the column where the solute concentration per unit of column height is low and further apart in those parts of the column where the solute concentration per unit of column height is high.

The apparatus and preferred modes of operation thereof will now be described with reference to the accompanying drawings, which are illustrative, in which:

Figure 1 shows the apparatus in elevation, partly in section.

Figure 2 shows a cross-sectional view of a preferred agitating disc.

Figure 3 shows a cross-sectional view of a preferred agitating disc.

Referring again to Figure 1, the column is provided with an inlet 4 at the top and an outlet 7 at the bottom. The column is also provided with further inlets 5 and 6 on the side and bottom respectively and a further outlet 3 at the top. The column is provided at top and bottom with disengaging spaces 11 and 12. Shaft 8 is vertically disposed throughout the central portion of the column and is externally connected to a drive mechanism 2, for example through a sliding coupling 16 and crank 15. The shaft is mounted in appropriate bearings 13 and 14 so that it operates in a vertical direction. The drive mechanism 2 imparts to shaft 8 a reciprocating motion in a vertical direction. Mounted on shaft 8 by means of hubs 10 are agitating discs 9, preferably at varying distances apart as illustrated in Figure 1. Cross-sectional views of two illustrative agitating discs are shown in Figure 2 and 3. If desired, the discs may be adjustable so as to vary their locations on the shaft 8. Inlet 5 is optional depending on the use to be made of the apparatus but it is preferred to provide the inlet and fit it with a valve or similar device for putting it out of operation.

Preferably the apparatus is provided with two or three inlets and two outlets. Preferably one inlet and one outlet is situated at the top of the column and one inlet and one outlet at the bottom of the column. Where a third inlet is used, this is provided between the top and bottom inlets, preferably at the midpoint of the column.

One preferred mode of operating the apparatus utilizes a feed solution and two

mutually immiscible solvents which have different specific gravities [hereinafter called the heavy or light solvent according to whether the one referred to has the higher or lower specific gravity]. The apparatus is operated by introducing the heavy solvent into column 1 through inlet 4 at the top. It flows downward through the column and is withdrawn through outlet 7 at the bottom. The light solvent is introduced through inlet 6 at the bottom of the column and flows upwardly through the column countercurrent to the heavy solvent and is withdrawn through outlet 3 at the top. The solution, which is to undergo extraction, is introduced through inlet 5 which is located between inlets 4 and 6, usually at about the midpoint of the column. The reciprocating motion of the shaft agitates the countercurrently flowing liquids so that there is formed a dispersion consisting of a continuous liquid phase and droplets of liquid dispersed throughout same and in intimate contact therewith.

Another preferred method of operating the apparatus utilizes a solution and one solvent which is immiscible therewith. The solution is introduced into the column 1 through inlet 4 or 6, depending upon its specific gravity compared to the solvent, and the solvent is introduced at the opposite end of the column. Inlet 5 is not used. The liquids which have passed through the column are withdrawn through outlets 3 and 7.

The spacing of the discs on the shaft and the speed and amplitude of reciprocation depend on the properties of the liquid system, e.g. interfacial tension, density differences, and they may vary within fairly broad limits. The discs may be mounted from one inch to six inches apart on the shaft. This spacing may vary within the column as shown in Figure 1. The shaft may be adjusted to reciprocate from 10 to 2000 times per minute, preferably from 50 to 1000 times per minute. The amplitude may be from 1/16 inch to 2 inches, preferably about 1/2 inch. In general, the speed of reciprocation varies inversely with the amplitude.

WHAT WE CLAIM IS:

1. An extraction apparatus for the purpose herein described, comprising a vertical elongated unbaffled column having inlets and outlets for introduction and removal of liquids, a vertical centrally located movable shaft adapted to reciprocate in a vertical direction, a plurality of horizontal open network agitating discs mounted on said shaft, each of said discs extending over substantially the entire interior cross-section of the column while leaving room for the passage of liquid between the periphery thereof and the interior wall of the column and containing large openings together comprising 50% to 65% of the cross-sectional area of the disc, and a reciprocating drive mechanism for said shaft.

2. An apparatus as claimed in claim 1, wherein said agitating discs each consist of expanded metal latticework.

3. An apparatus as claimed in claim 1, wherein said agitating discs each consist of a spoked wheel-like plate.

4. An apparatus in accordance with claim 2 or claim 3, wherein the number of openings in said discs is the minimum consistent with the requirements of disc strength.

5. An apparatus in accordance with any one of the preceding claims, wherein two inlets and two outlets are provided.

6. An apparatus in accordance with any one of the preceding claims, wherein there is provided a liquid inlet and a liquid outlet at the top of the column and a liquid inlet and a liquid outlet at the bottom of said column.

7. An apparatus in accordance with claim 6, wherein a further liquid inlet is provided between said inlets, preferably at the mid-point of said column.

8. An apparatus in accordance with any one of the preceding claims, wherein a disengaging space is provided at the top and at the bottom of said column.

9. An apparatus in accordance with any one of the preceding claims, wherein the said reciprocating drive mechanism is a variable reciprocating drive mechanism.

10. An extraction apparatus for countercurrent extraction of liquids substantially as described with reference to the drawings.

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876,070 COMPLETE SPECIFICATION
1 SHEET



FIG. 1

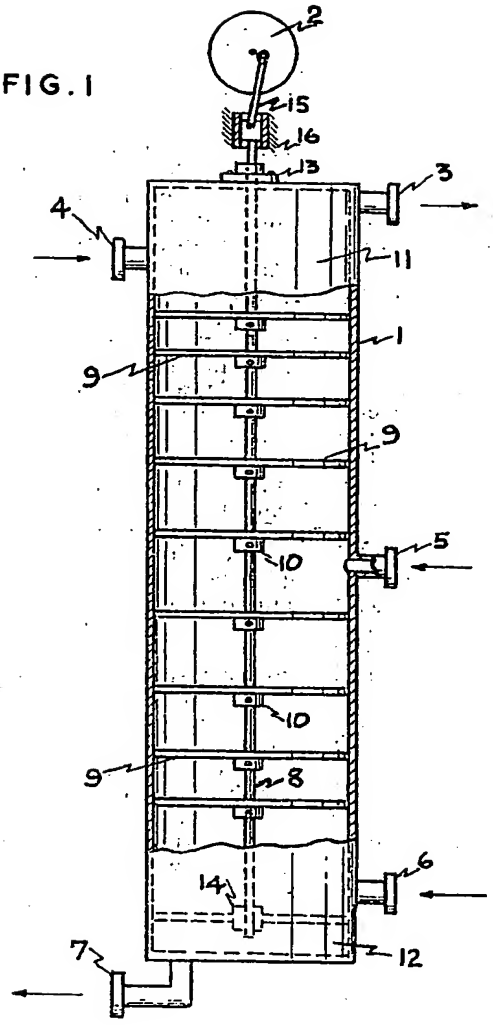
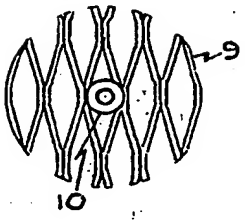



FIG. 2



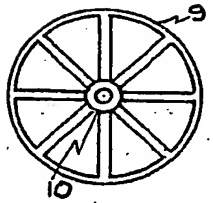




FIG. 3